Final Report

AOARD-09-4037
(FA2386-09-1-4037)

Title:
Development of nano processing technology for shape memory alloy fibers

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Jan.30, 2011

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Report Documentation Page

Form Approved
OMB No. 0704-0188

1. REPORT DATE	2. REPORT TYPE	3. DATES COVERED
09 FEB 2011	**Final**	**06-03-2009 to 01-09-2010**

4. TITLE AND SUBTITLE
Development of nano processing technology for shape memory alloy fibers

5a. CONTRACT NUMBER
FA23860914037

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)
Hiroyuki Kato

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Hokkaido University,N13W8 Kita-ku,Sapporo 060-8628,Japan,NA,NA

8. PERFORMING ORGANIZATION REPORT NUMBER
N/A

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
AOARD, UNIT 45002, APO, AP, 96338-5002

10. SPONSOR/MONITOR'S ACRONYM(S)
AOARD

11. SPONSOR/MONITOR'S REPORT NUMBER(S)
AOARD-094037

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution unlimited

13. SUPPLEMENTARY NOTES

14. ABSTRACT
The effect of processing conditions on the preparation of nano NiTi SMA (shape memory alloys) fibers were examined in this study. Firstly, of alumina were prepared The holes in the nano porous membranes were 200nm in diameter. 50.3%Ni-Ti alloy was sputtered on the membrane, and columnar crystals having the diameter close to the holes were grown. The columnar crystals were broken into pieces by ultrasonic waves. The martensitic transformation was confirmed to occur in the disintegrated nano crystals of the SMA. DSC curves show that the martensite transformation temperatures and latent heat of the nano crystals are equal to those of the bulk state 50.3%Ni-Ti alloy.

15. SUBJECT TERMS
Shape Memory Alloys, Smart Structures

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			
unclassified	**unclassified**	**unclassified**	**Same as Report (SAR)**	**6**	

Abstract

The effect of processing conditions on the preparation of nano NiTi SMA (shape memory alloys) fibers were examined in this study. Firstly, of alumina were prepared The holes in the nano porous membranes were 200nm in diameter. 50.3%Ni-Ti alloy was sputtered on the membrane, and columnar crystals having the diameter close to the holes were grown. The columnar crystals were broken into pieces by ultrasonic waves. The martensitic transformation was confirmed to occur in the disintegrated nano crystals of the SMA. DSC curves show that the martensite transformation temperatures and latent heat of the nano crystals are equal to those of the bulk state 50.3%Ni-Ti alloy.

Background

This study is a part of the collaborative work "Development of nano SMA/SMP fiber composite material and applications", with U of Washington, U of British Columbia and Gifu University, Japan, teams. In this work I am responsible for supplying nano SMA fibers to the other members in collaboration, who will utilize the fibers for further processing.

As the result of the AOARD project last year, I prepared alumina templates having nano holes on the surface (Fig.1). By conventional argon ion sputtering, 50.3at.%nickel-titanium SMA was sputter-deposited on the membrane. Figure 2 is the as-deposited NiTi metallic film on the alumina membrane.

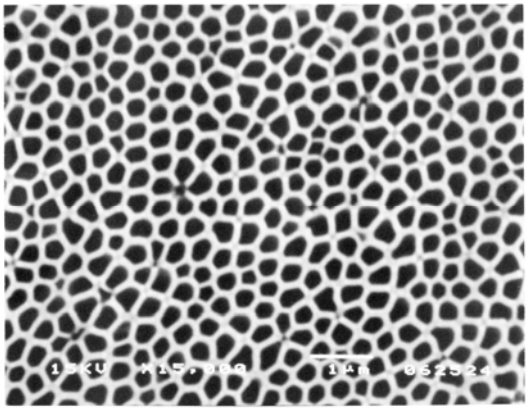

Fig.1 Nano porous alumina membrane.

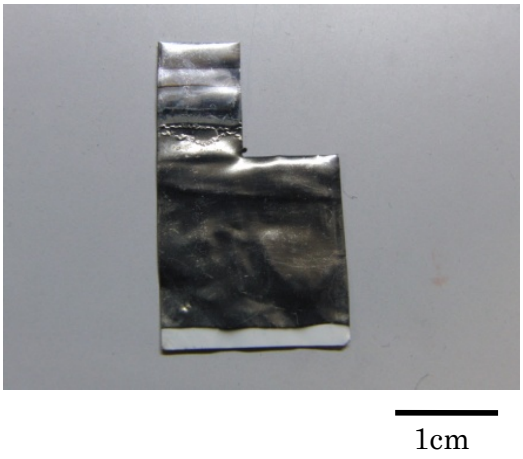

Fig.2 Columnar sputtering product of NiTi.

Result of this project

Under appropriate sputtering conditions, columnar crystals were grown on the alumina, as shown in Fig. 3. This year, I intended for finding the optimum condition for growing metallic NiTi thin film of desired composition on the porous alumina membrane. The arrow indicates the direction of growth of the columnar crystals.

This film was obtained by sputtering for 30min by adjusting the substrate temperature and argon flow. Under the same condition, the prolonged sputtering for 1h was performed, and a film twice as thick as this film, approximately 4μm, was obtained.

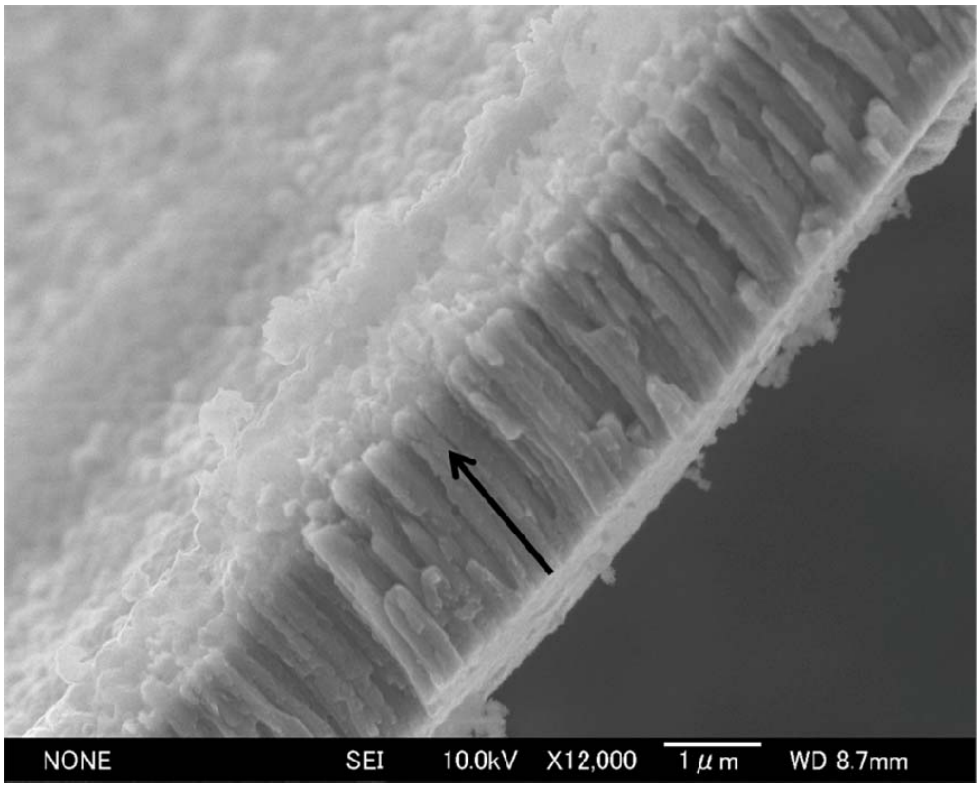

Fig.3. The cross section's view. Arrow indicates the direction of crystal growth.

Figure 4 shows X-ray diffraction patterns of the sputtered film before and after annealing at 500C for 1hr. The pattern before annealing has no Bragg peak, indicating that the film was not crystallized. After the annealing was done, some sharp Bragg peaks are seen in the pattern; the peak positions are confirmed to be the same positions of those of bulk NiTi alloys.

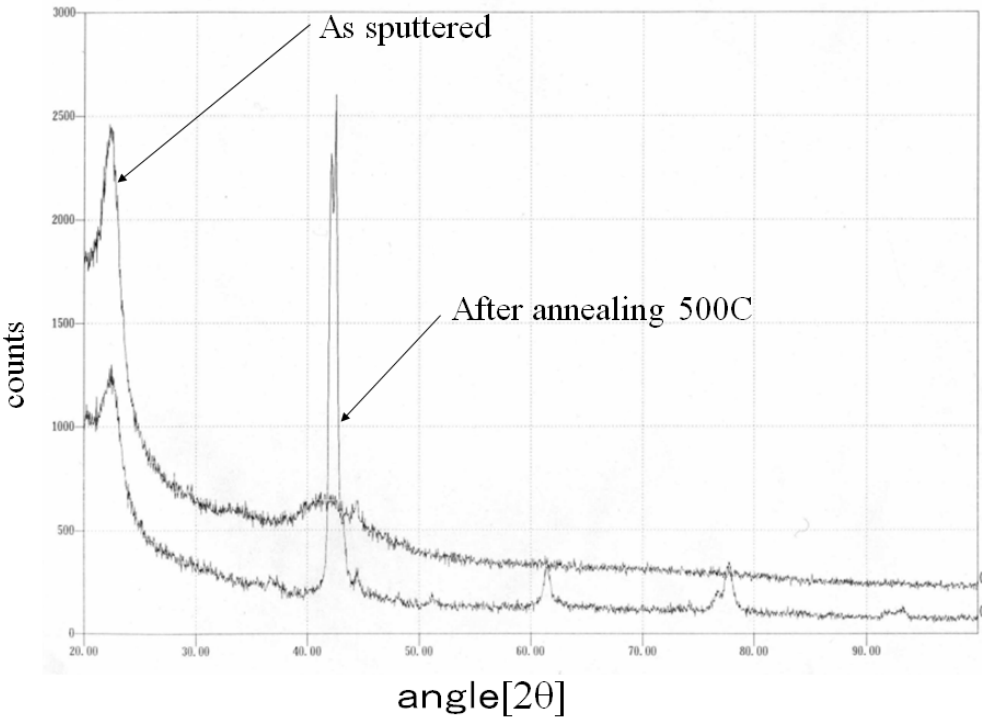

Fig.4 X-ray diffraction patterns of the as-sputtered and annealed film. As sputtered film is not crystallized, and the film after annealing at 500C shows some sharp Bragg beaks indicating that it has been crystallized.

This study has found that the sputtered film is very fragile; it is easy to break it into nano-size pieces by operating conventional ultrasonic washing machine. Figure 5 shows the ethanol-metallic fiber mixture after the ultrasonic disintegration.

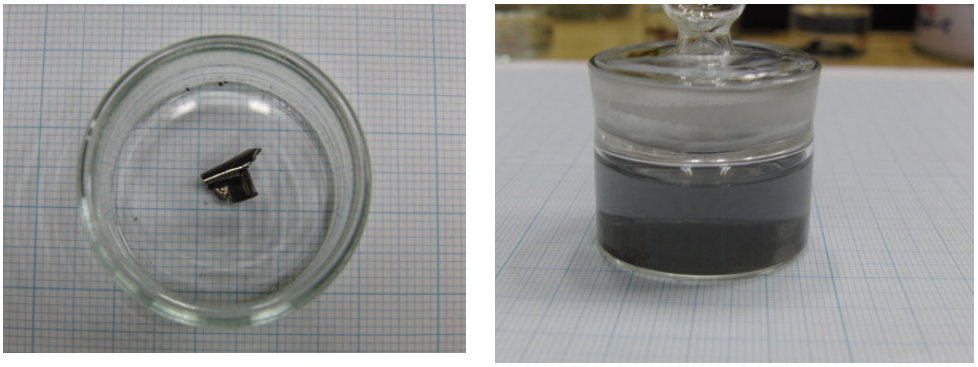

Fig.5 The metallic film (left) was broken into Nano crystals (right) by applying ultrasonic wave.

Then, a mesh for transmission microscope was immersed in the mixture, and the disintegrated fibers were observed by the electron microscope. The picture is shown in Fig.6.

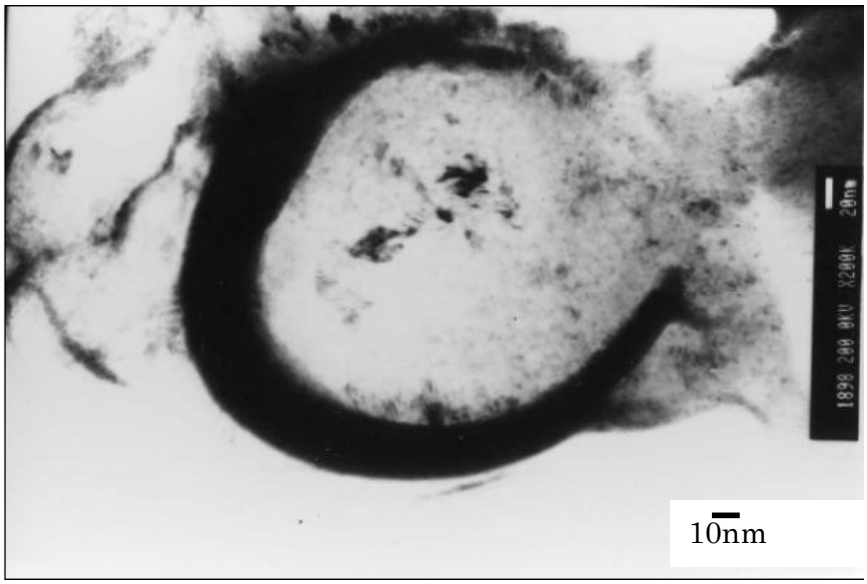

Fig.6 TEM micrograph of the isolated fibers after ultrasonic disintegration of the sputtered NiTi film.

Finally, the martensitic transformation of the nano fibers was observed by differential scanning calorimeter (DSC). The DSC curves are shown in Fig.7. The curves show peaks indicating heat absorption and emission occurs in the nano fibers. The temperatures of the peaks and their height are almost equal to the curves of bulk NiTi alloys, so that it is sure that the martensitic transformation occurs in nearly 100% volume of the annealed film.

Publications

1. A part of this report will be presented on the US/Japan workshop of reconfigurable materials, sponsored by AFOSR, organized by Drs. Les-Lee and M. Taya, held in Sep.2010, Sapporo JAPAN.
2. A part of the present result will be presented the international conference on "Martensitic transformations", Sep.4-9, 2011. Osaka, Japan.
3. A scientific paper is now under-preparation and will be submitted to some scientific Journal.

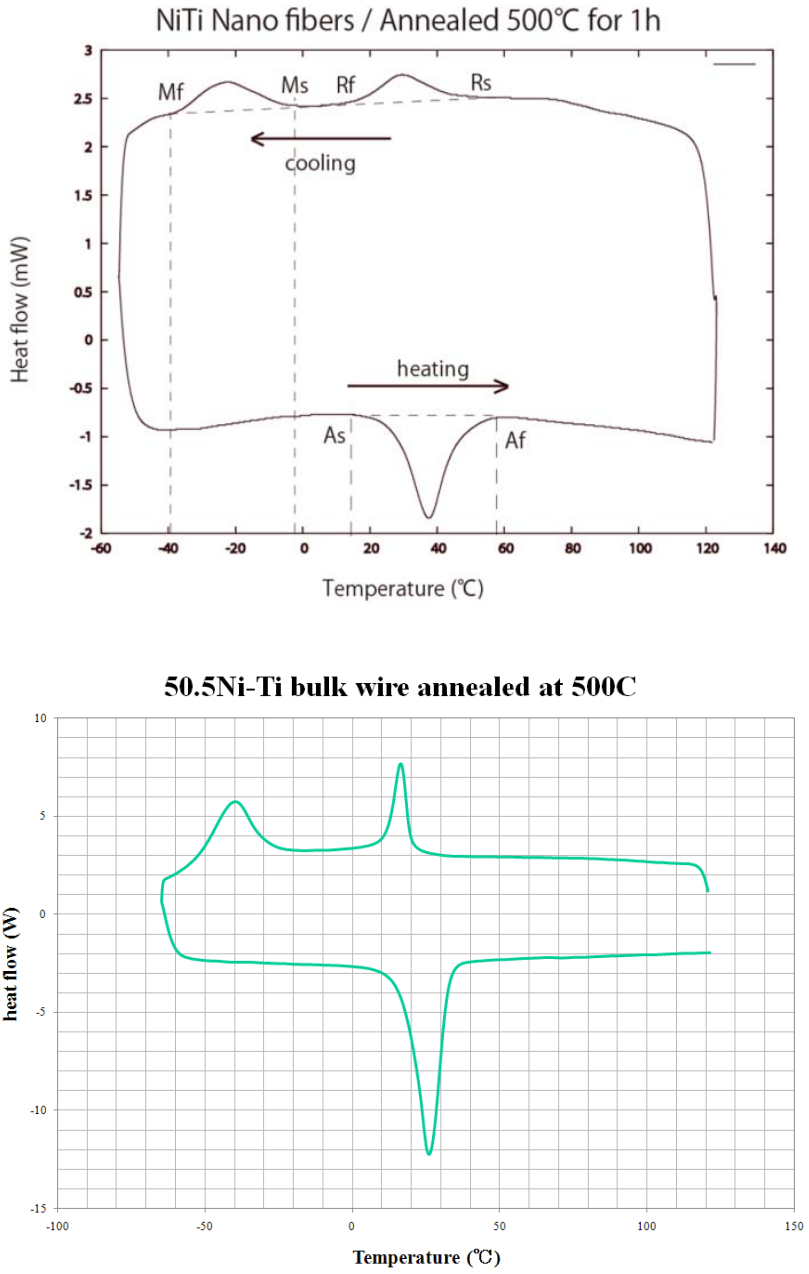

Fig.7 Differential scanning calorimeter curve of the Nano fiber (upper) and typical bulk metal (lower). The curves have both endothermic and exothermic peaks indicating martensitic phase change occurred there. The amount of heat is equal between the Nano fibers and the bulky alloy, so it assures the Nano fibers transform almost 100% in volume. EOF